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New Quinone-Based Pulping Catalysts

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NEW QUINONE-BASED PULPING CATALYSTS

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ABSTRACT

2,6/7-Dimethylanthraquinone (DiMAQ) and its precursor, octahydro-DiMAQ (ODiMAQ), have been synthesized from benzoquinone in high yield and at potentially low cost. Both catalysts were twice as active as anthraquinone in soda, kraft, and polysulfide pulping systems. Pulps produced using these catalysts were as easy to bleach and had the same strength properties as the corresponding AQ pulps. Analysis of unbleached pulps indicated that ODiMAQ was converted to DiMAQ during pulping and that the latter was approximately four times higher in amount than the residual AQ, suggesting that the end-of-cook concentration of DiMAQ may be higher than that of AQ.

INTRODUCTION

The addition of catalytic amounts of anthraquinone (AQ) to alkaline pulping processes offers several advantages, including increased pulping rates, higher pulp yields, and improved capacity of the chemical recovery system [1]. The increased use of AQ in kraft pulping systems in recent years is probably related to a substantial reduction of its price [2]. We have been investigating ways to prepare pulping catalysts at an even lower cost and, thus, encourage more widespread use of catalysts [3]. We have prepared AQ-type catalysts by treating a hardwood lignin with an oxidizing agent to produce 2,6-dimethoxybenzoquinone (Figure 1), which is then converted into 2,6/7-dimethylanthraquinone (DiMAQ) precursors and other structurally related compounds by treatment with isoprene (a Diels-Alder reaction). Many of these precursors are converted to DiMAQ by loss of methanol and hydrogen during the catalyst preparation (Figure 1); others are converted to DiMAQ during the cooking process. This method of preparing catalysts has the advantage of utilizing an inexpensive starting material, lignin; however, it is hampered by low synthetic yields. The first (oxidation) step is best accomplished by treating a hardwood lignin with nitrogen dioxide; the yield is only ~10% [4]. The addition of a diene, the second synthetic step, gives a mixture of components, the major one being DiMAQ in ~40% yield.

This report (a) outlines a new, efficient, and potentially low cost synthesis of an effective pulping catalyst from benzoquinone, (b) compares the pulping activity of DiMAQ and related structures to that of AQ, and (c) examines the bleachability and strength of DiMAQ pulps, relative to that of AQ pulps. Of particular interest is the preparation and pulping activity of octahydro-DiMAQ (ODiMAQ), the synthetic precursor of DiMAQ. In addition, data is presented on the pulping activity of the Diels-Alder reaction product (referred to as the "catalyst mixture" herein) obtained from dimethoxybenzoquinone and isoprene (Figure 1).

CATALYST PREPARATION FROM BENZOQUINONE

The Diels-Alder reaction of benzoquinone (BQ) with isoprene is an alternative way to prepare DiMAQ. Yield optimizations were conducted by varying the reaction solvent, temperature, time, and reactant ratios. With ethanol as the solvent, a mixture of bis-adduct isomers, ODiMAQ, can be obtained in high yields (Figure 2). [With toluene

as the solvent, ODiMAQ is obtained in 68% yield, along with a monoadduct product in 30% yield.] The best synthetic conditions involved thoroughly mixing a 2:1 ratio of isoprene to BQ in absolute ethanol for 2 hours at 165°C. Gas chromatography (GC) indicated a 93% yield of ODiMAQ; the isolated yield from 100 g scale runs was routinely ~85%. Treatment of the ODiMAQ with oxygen and KOH in an organic solvent gave DiMAQ in 93%-isolated yield (Figure 2). However, this additional synthetic step is unnecessary; the pulping activity of ODiMAQ is the same as DiMAQ (see below).

In contrast to our earlier work, this process provides a direct route to a pure, effective pulping catalyst (ODiMAQ) in high overall yield, and uses less isoprene than the catalyst-from-lignin route. The process requires a more expensive starting material (benzoquinone vs. lignin). Preliminary cost estimates indicate that the cost of ODiMAQ will be similar to that of AQ. However, since ODiMAQ is twice as active as AQ (see below), its effective cost would be significantly lower than that of AQ.

PULPING STUDIES

We conducted about 50 small-scale pulping studies involving 50 g of southern pine chips. The results indicate that DiMAQ and ODiMAQ were twice as effective as AQ in catalyzing delignification in soda-, kraft-, and polysulfide-catalyst cooks. This two-fold increase in reactivity was observed at comparable AQ:DiMAQ levels of 0.1:0.05% and 0.05:0.025% on wood. For larger scale (1 kg) cooks of southern pine a comparison was made only at the 0.1/0.05 levels. Table 1 presents the large-scale cook results. The reproducibility of the cooks was quite good, as can be seen by the five soda/AQ cooks that were conducted. As was the case in the small-scale runs, the new catalysts at half the doses performed as well as full doses of AQ for producing 30 kappa pulps for each of the three pulping systems. Also, as expected [5], the presence of a catalyst improved the yield in the kraft case.

The value of the catalyst in kraft systems can be seen from the higher kappa number (37.5) of the standard control kraft cook that has no catalyst, but was performed under the same conditions as the kraft/catalyst cooks. A 1-kg soda control was not performed; however, based on previous small-scale experiments, we expect the kappa number would have been about 60. Two kraft cooks were performed at elevated H-factors to get a 31-kappa pulp for comparison to the kraft/catalyst pulps; the resulting 28.1 and 33.5 kraft pulps were combined to give the desired 31-kappa pulp.

The pulps were extracted and the level of catalyst determined by GC. The data obtained (Table 1) indicate that (a) ODiMAQ was aromatized to DiMAQ during cooking and (b), except in the polysulfide cooks, the level of recovered catalyst was ~4x higher for DiMAQ and ODiMAQ than for AQ. The latter result may explain the superior effectiveness of the new catalysts; the DiMAQ concentration may be higher throughout the cook than AQ.

The "catalyst mixture" led to similar delignification levels as the other DiMAQ (or ODiMAQ) cooks; however, the pulp yield and viscosity were somewhat lower in the catalyst mixture cases. Because of limited availability of the catalyst mixture, we were unable to perform multiple cooks to confirm these trends.

BLEACHING STUDIES

The "bleachabilities" of DiMAQ-related pulps were compared to the corresponding AQ pulps. Bleachability refers to the amount of bleaching chemicals consumed to reach a certain brightness for a set of pulps. This brightness can be the final brightness or some intermediate brightness. By going to fully bleached pulps, we were able to compare brightness values at many points and generate pulps for strength measurements. The bleachability of the pulps was determined using a D(EOP)DED bleaching sequence, in which D = chlorine dioxide (ClO_2), E = extraction with dilute NaOH, and EOP = extraction with dilute NaOH in the presence of oxygen and hydrogen peroxide (H_2O_2).

The bleaching results are shown graphically in Figures 3 and 4. Duplicate bleaching runs on the same pulp and on two pulps produced by the same catalyst showed good reproducibility. The small differences observed in brightness and viscosities between pulps produced with different catalysts were within the experimental error for duplicate runs

performed on the same pulp. On this basis, we concluded that the kraft and kraft/catalyst pulps were equally bleachable; the same held true for the soda/catalyst pulps.

The slightly lower amount of chlorine dioxide consumed in the soda/AQ and kraft/AQ cases hints that DiMAQ pulps may be slightly harder to bleach. The lower viscosity of the kraft control may reflect the longer cooking times that were required to reach the targeted kappa number. A longer cook would likely exhibit more carbohydrate damage.

The kraft pulps produced from the catalyst mixture were a little more difficult to bleach; however, the soda pulps produced using the catalyst mixture showed no differences in brightness, viscosity, and chlorine dioxide consumed from that shown by the other pulps.

PULP STRENGTH COMPARISONS

The fully bleached pulps (starting freeness of ~750) were refined in a PFI mill at 10% consistency to provide pulps at roughly three different levels of freeness: 300, 400, and 600. Handsheets made from the beaten and unbeaten pulps were then evaluated for specific strength parameters. Table 2 compares the average density, burst, and zero-span values for the nine pulps at 350 and 500 Canadian standard freeness (CSF); viscosity values for the unbeaten pulps are also provided in the table. As expected, lower freeness values normally led to increased values of density, burst, and zero-span. The latter, which is a measure of individual fiber strength, should correlate with the viscosity, a measure of average chain polymer length. Except for an anomalously low viscosity for the kraft control, the data in the table indicate no real differences in viscosities and zero-span indexes. In general, the strength values in Table 2 are all within the standard deviation (± values in the table) of one another within the two series.

Strength comparisons of two carefully prepared soda/AQ and DiMAQ pulp handsheets are shown in Figures 5-9. Property trends are nearly identical for the two pulps for each of the strength parameters examined. Another frequently used measure of comparative strength properties involves comparing plots of tear index vs. tensile index for each pulp (Figure 5); here again, there was no basic differences between the soda/AQ and DiMAQ pulps. Since ODiMAQ converts to DiMAQ during cooking, extensive testing of ODiMAQ pulps were not performed; a limited number of handsheets showed properties nearly identical to that of the AQ and DiMAQ pulps.

Within the experimental error of our measurements, the kraft and kraft/catalyst pulps were very similar with regards to tear index vs. tensile index trendlines over the span of freenesses examined (Figure 10). The trendlines shown in Figure 10 are based on 80 data points. The expected downward slope in the trendlines is apparent. [Data for kraft/DiMAQ pulp tear vs. tensile are not included here because of a problem with the preparation of some of the handsheets; limited results suggest no difference from the other data.] The addition of DiMAQ, ODiMAQ, or AQ to a kraft pulping system had no adverse effects on tear/tensile strength parameters; this was expected for the kraft/AQ case [5].

SUMMARY

A new, simple, 2-step, high-yield preparation of DiMAQ from benzoquinone has been developed. The product of the first step, octahydro-DiMAQ, is a very effective pulping catalyst, as good as DiMAQ itself, and twice as good as AQ. The pulps produced from the new catalysts are as easy to bleach and are as strong as AQ pulps. Consequently, we now have a one-step, high-yield, economical preparation of a new, effective pulping catalyst. Steps are being taken to commercialize octahydro-DiMAQ.

EXPERIMENTAL

Preparation of octahydro-2,6/7-dimethylanthraquinone (ODiMAQ). A mixture of benzoquinone (0.45 mole) and isoprene (1.13 mole) in ethanol (100 mL) was sealed in a Parr 300-mL stainless steel bomb and heated at 170°C for 7 hours. This produced a pressure of 170 psi. After cooling, the bomb contents were cooled, filtered, and washed with cold ethanol to afford a white solid. Successive concentrations and precipitations produced additional batches of solid. A yield of 80% was found in the first fraction. An additional 5% could be isolated from the filtrate upon concentration.

Analysis of the collected ODiMAQ by GC/MS indicated the presence of three compounds, eluting very near one another, with identical molecular weights *m/z:* 244 (M^+). Several isomers are possible, based on the geometry of the methyl groups and the nature of the ring conjunctures relative to each other. The structures of the ODiMAQ products were confirmed by ^1H NMR ($CDCl_3$) d 5.37 (bs, 2H, =CH); 3.03 (apparent q, J=6 Hz, 2H, CHC=O); 2.96 (apparent q, J=6 Hz, 2H, CHC=O,); 2.51-2.34 (m, 4H, CH_2-C=); 2.22-2.08 (m, 4H, CH_2-C=); 1.77 (bs, 6H, CH_3).

Preparation of 2,6/7-dimethylanthraquinone (DiMAQ) from ODiMAQ. Attempts to oxidize ODiMAQ to DiMAQ using basic hydrogen peroxide and chlorine dioxide failed; however, ODiMAQ was oxidized by two other methods. First, a suspension of ODiMAQ (10.5 mmol) in 100 mL of ethanol containing KOH (35.7 mmol, 3.40 equivalent) was heated at reflux under a stream of air. The mixture turned dark green and then dark red after heating for 1 h. After heating for an additional 3 h, the mixture was cooled, stirred at room temperature overnight, acidified to pH 1, and filtered. Partitioning between ether and water purified the collected crude product. Evaporation of the ether solvent under reduced pressure gave DiMAQ (8.52 mmol, 81%). The sample could be further purified by recrystallization (EtOAc/hexane). Analysis by GC showed two DiMAQ isomers of nearly identical retention time. The presence of DiMAQ was confirmed by comparing spectral properties to previously prepared DiMAQ [3]. ^1H NMR showed: ($CDCl_3$) d 8.19 (d, J=8 Hz, 2H, H-4 + H-8 or H-5); 8.09 (d, J=1 Hz, 2H, H-1 + H-5 or H-8); 7.58 (dd, J=1, 8 Hz, 2H, H-3 + H-7 or H-6); 2.53 (s, 6H, $ArCH_3$); mass spectrum displayed a strong molecular ion at *m/e* 236.

The oxidation was also accomplished by stirring ODiMAQ (32.8 mmol) in 95% ethanol (400 mL) containing KOH (321 mmol) in a Parr 1000-mL stainless steel bomb with a glass liner under an O_2 atmosphere (145 psi) at room temperature. As ODiMAQ was added to the colorless caustic solution, the color changed to dark green. When the reaction was complete (no longer a green or red color, 2 h), a heterogeneous mixture was obtained which had a tan solid suspended in a clear solution. The mixture was diluted with water (400 mL) and acidified with concentrated HCl to pH 1. The mixture was filtered and the yellow/purple solid was suspended in 400 mL of methanol and filtered again. The solid was then dried under high vacuum and gave pure DiMAQ (30.5 mmol, 93%). Attempts to carry out the same oxidation in water failed to give any reaction, presumably due to poor adduct water-solubility. Oxidations in 75% ethanol/water were slower - mixtures of DiMAQ and ODiMAQ were observed after two hours.

Pulping Procedure. Softwood pine chips, obtained from a SE pulp mill, were screened using a KMW screen with the +1/4 to -1 inch fractions retained and then separated by hand to remove any bark. Chips were stored frozen prior to use. The percent solids of each chip bag was determined by drying samples for at least 24 h in a 105°C oven. Solutions of NaOH, Na_2S, and polysulfide were prepared to be approximately 110-120 g/L as Na_2O. The NaOH and Na_2S concentrations were determined according to the ABC titration procedure [6]. The PS solutions were titrated according to procedures outlined in TAPPI procedure T 694. Large-scale cooks were performed at a 4:1 liquor:wood ratio on wet chips which, if oven-dried (o.d.), would weigh 1 Kg. The H-factor values [7] of the large-scale cooks were determined using a cook programmer.

Soda Pulping	Kraft Pulping	Polysulfide Pulping
18% NaOH (as Na_2O)	18% active alkali	19% active alkali
15 min from 80 - 100°C	25% sulfidity	12 % sulfidity
90 min from 100 - 170°C	15 min @ 100°C, 60 min from 100 - 170°C	1.22 % PS
120 min @ 170°C	70 min @ 170°C	60 min 100 - 170°C
Target H-factor: 2100	Target H-factor: 1200 for catalyst cooks	70 min @ 170°C
	Target H-factor: 1400 and 1350 for control cooks	Target H-factor: 1050

The product from each cook was disintegrated and then washed with water. The resulting pulp was screened using a Sprout-Waldron, 0.01 inch slot screen apparatus, centrifuged, weighed, and fluffed. The pulp was weighed before and after drying in a 105°C oven for at least 24 hours. The ratio of dry to wet sample is the percent consistency. Percent yield was calculated from the following equation: % Yield = (% CSY of the pulp)(g of pulp)/(g o.d. chips used in digester) x 100%. The pulp was analyzed for kappa number according to TAPPI procedure T236 and for viscosity according to TAPPI procedure T230.

Percent Residual Catalyst. Exactly 5.0 g o.d. pulp was air dried, placed in approximately 250 mL $CHCl_3$ that contained 2.00 mL of 0.0642 mg/mL 2,3-DMAQ standard (std.) solution, evaporated to dryness using a rotary evaporator, and extracted with $CHCl_3$ for 24 hours in a Soxhlet extraction apparatus. The resultant $CHCl_3$ solution was evaporated to a small volume, transferred to a 2 mL vial, and then evaluated by gas chromatography (GC) for percent residual catalyst using the following calculation:

$$\%\ \text{residual catalyst} = \frac{(\text{cat. peak area})(\text{mg std. added})(\text{response factor})}{(\text{std. peak area})(\text{mg of cat. used in cook})} \times \frac{(\text{g of o.d. pulp yielded in cook})(100)}{(\text{g of o.d. pulp extracted})}$$

Bleaching. All stages used varying amounts of 10% consistency (CSY) pulp.

- D_0 - 0.2 kappa factor (KF), 50°C, 45 min
- EOP - % NaOH = Total Active Chlorine (TAC = KF x kappa #) x 0.6, 70°C, 60 min, 0.5% H_2O_2, 60 psig O_2 pressure → 0 psig
- D_1 - 70°C, 90 min, 1.0% ClO_2, and 0.25% caustic
- E_2 - 70°C, 60 min, 0.5% caustic
- D_2 - 70°C, 180 min, 0.2% ClO_2

Each sample was evaluated for its kappa number after the EOP stage using TAPPI procedure T236, for its ISO brightness reading after its EOP, D_1, E_2, and D_2 stages using TAPPI procedure T534, for filtrate % residual ClO_2 after each D stage using CPPA method J14P, and for filtrate pH after each bleach stage.

Strength Tests. The nine fully bleached pulp samples of interest were evaluated for viscosity using TAPPI procedure T230. These samples were also refined in a PFI mill to three different freeness points. Handsheets were prepared (TAPPI procedure 205) for each refined and unrefined bleached pulp and the following strength tests (TAPPI procedure T220) determined: basis weight, density, tensile, tear, zero-span tensile, and burst.

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Table 1. Detailed Pulping Results for 1-Kg Pine Cooks[a]

Cook Type	Catalyst Type	% Catalyst Applied	Cook H-factor	Kappa Number	Screened Yield (%)	Viscosity mPa•s	Recovered Cat. - %[b]
Soda	AQ	0.10	2090	33.7	45.0		
		0.10	2097	33.1	43.7		
		0.10	2089	33.1	45.1		
		0.10	2111	31.2	45.4		
		0.10	2114	32.3	44.1	17.3	0.3
	ODiMAQ	0.05	2124	33.6	45.6	18.2	0.9
		0.05	2106	30.8	43.5	16.0	
	DiMAQ	0.05	2106	33.7	45.4	18.5	1.8
		0.05	2108	35.2	45.6	18.1	
	cat. mixture[c]	0.05	2099	34.0	43.9	16.0	2.0
Kraft	AQ	0.10	1214	30.3	46.8	36.7	
		0.10	1192	31.6	47.3	38.2	0.5
	ODiMAQ	0.05	1195	31.8	46.1	40.2	1.9
		0.05	1200	30.8	47.0	36.8	
	DiMAQ	0.05	1197	32.7	47.2	36.4	
		0.05	1197	31.3	46.5	37.7	2.3
	cat. mixture[c]	0.05	1197	30.8	40.6	33.1	2.5
	none	0.00	1196	37.5	44.1	39.1	
	none[d]	0.00	1389	28.1	45.8		
	none[d]	0.00	1346	33.5	45.6	37.9	0.0
Polysulfide[e]	AQ	0.05	1053	32.2	46.4	29.6	0.3
		0.05	1040	33.0	46.7	31.4	0.1
	DiMAQ	0.025	1045	33.0	45.2	31.2	0.3
		0.017	1031	35.9	46.3	31.0	0.4

[a] Unless stated otherwise, the cooks were performed with an active alkali = 18%, sulfidity = 25% for kraft cases, and a final cook temperature = 170°C.

[b] The residual catalyst in the pulp was recovered by solvent extraction; % of applied catalyst.

[c] The dose employed was based on the amount of DiMAQ in the mixture.

[d] The H-factor was modified in an attempt to get a 31 kappa pulp; pulps were combined for viscosity measurement and further studies.

[e] PS = 1.22%, active alkali = 19%, sulfidity = 12%.

Table 2. Comparison of Selected Strength Parameters for Soda- and Kraft-Catalyst Bleached Pulps.

	Density g/cm³ (± 0.03)		Burst Index (kPa•m²/g) (± 0.5)		Zero Span Index N•m/g (± 10)		Viscosity mPa•s
CSF	350	500	350	500	350	500	
Kraft (no catalyst)	0.69	0.66	7.2	6.2	132	125	19.7
Kraft AQ	0.70	0.69	8.5	7.8	117	124	23.0
Kraft DiMAQ	0.67	0.67	8.4	7.8	118	122	21.1
Kraft DiMAQ (II)	0.73	0.71	7.6	7.1	128	117	22.7
Kraft ODiMAQ	0.70	0.69	7.3	6.3	116	110	24.5
Kraft Cat. Mixture	0.74	0.64	6.6	6.2	126	133	22.6
Soda AQ (II)	0.70	0.69	6.7	6.0	123	118	14.2
Soda DiMAQ	0.71	0.69	6.5	6.1	125	122	13.5
Soda DiMAQ (II)	0.70	0.68	6.7	6.2	127	116	12.7
Soda ODiMAQ	0.69	0.65	6.8	7.0	108	118	14.2
Soda Cat. Mixture	0.69	0.68	6.5	5.5	112	111	14.0

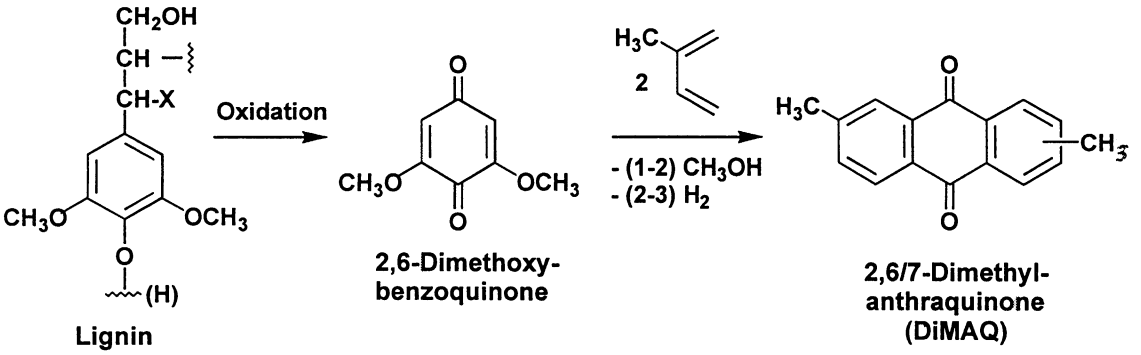

Figure 1. Chemical steps in the conversion of a hardwood lignin to DiMAQ.

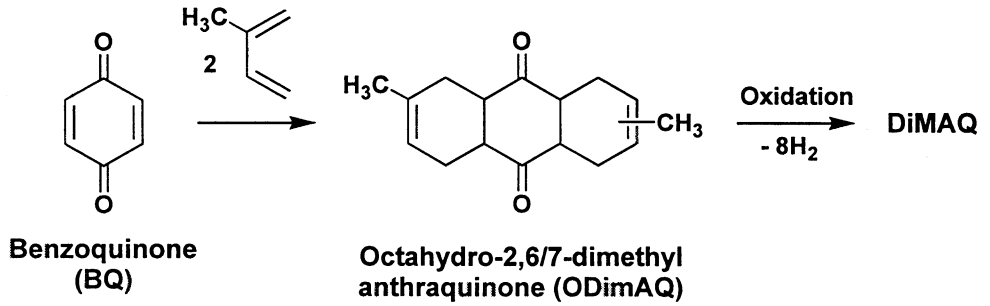

Figure 2. Preparation of DiMAQ from Benzoquinone.

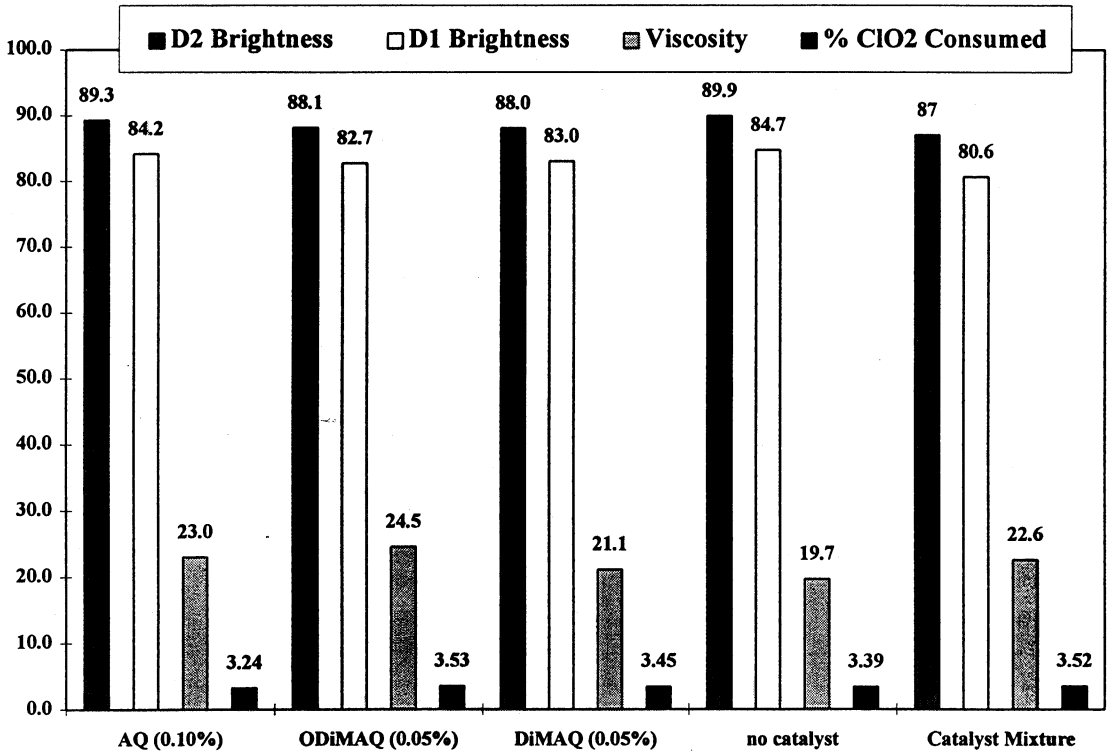

Figure 3. Kraft Bleaching Comparisons.

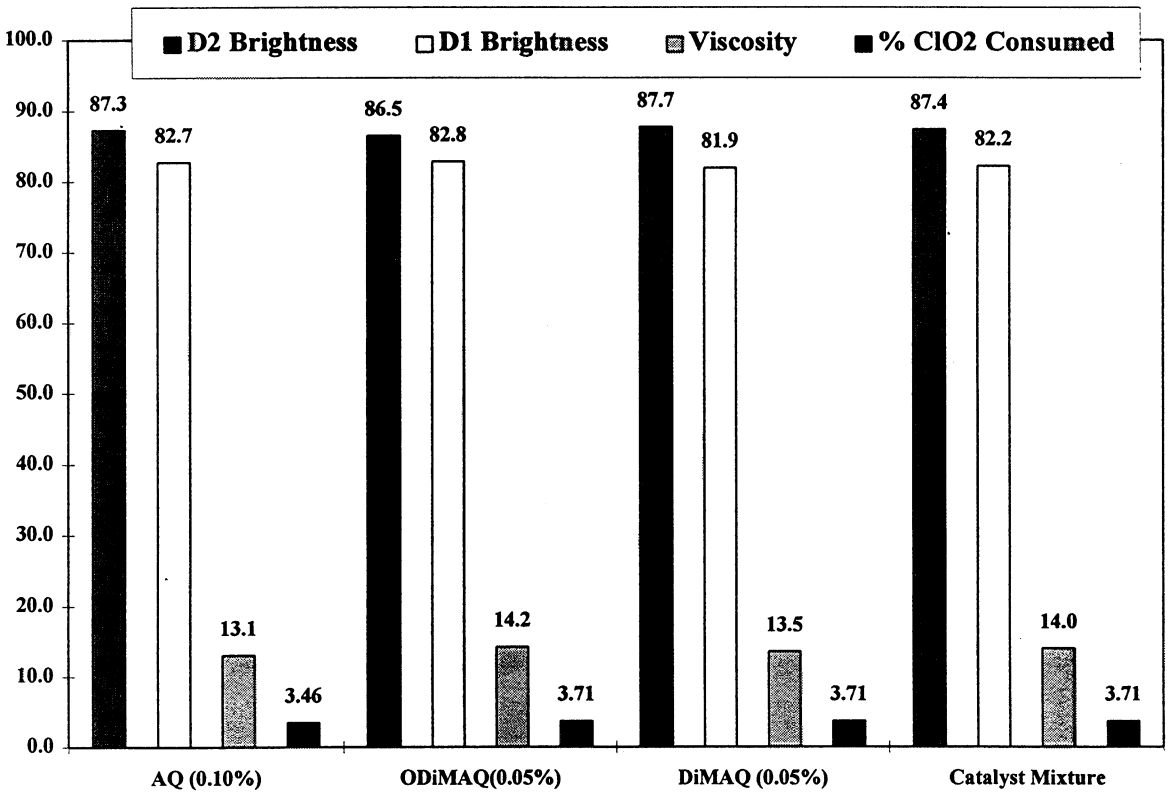

Figure 4. Soda Bleaching Comparisons.

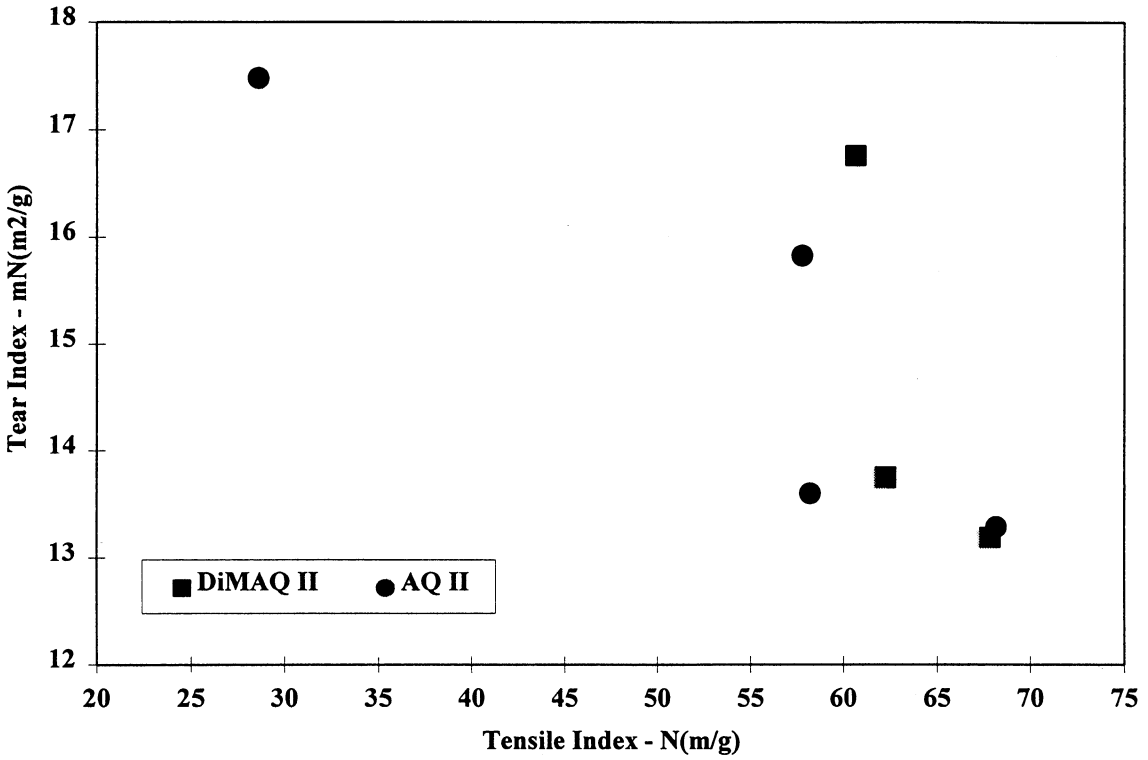

Figure 5. Comparison of Tear/Tensile Indexes for Two Soda/Catalyst Pulps.

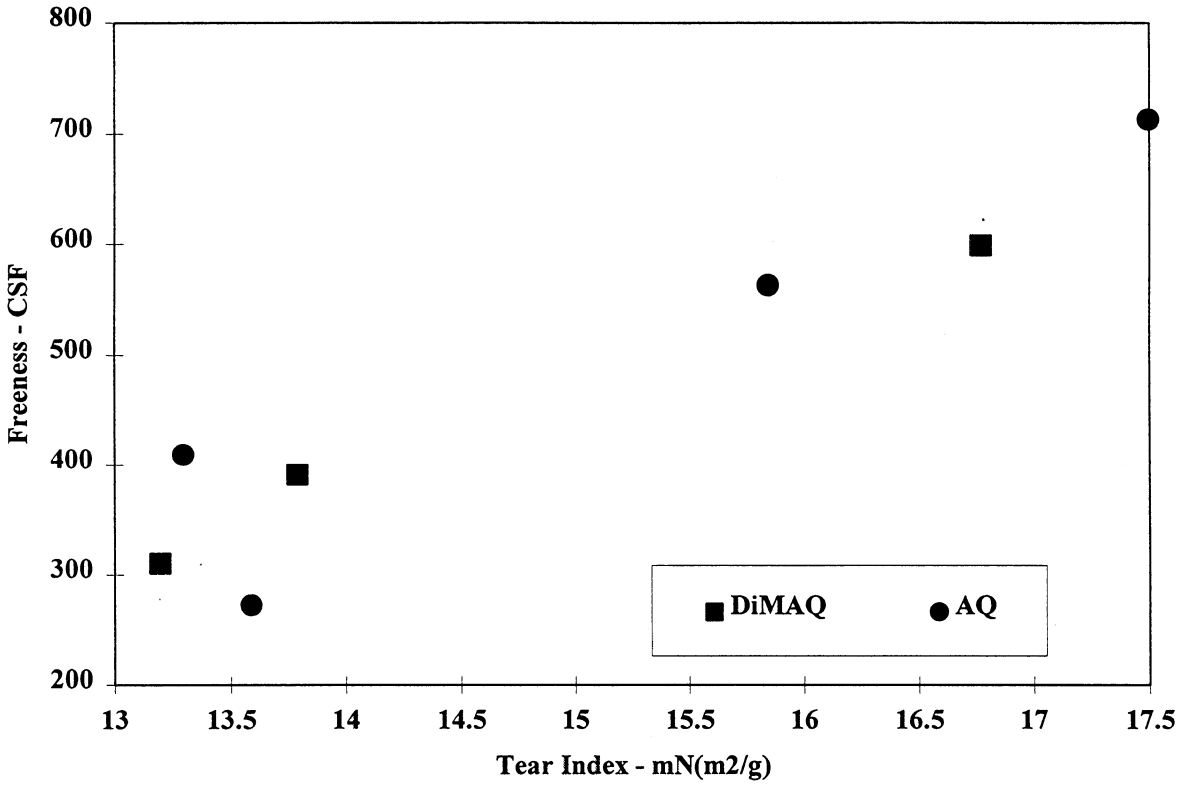

Figure 6. Comparison of Average Tear Indexes for Soda-AQ and Soda-DMAQ Pulps at Several Freenesses.

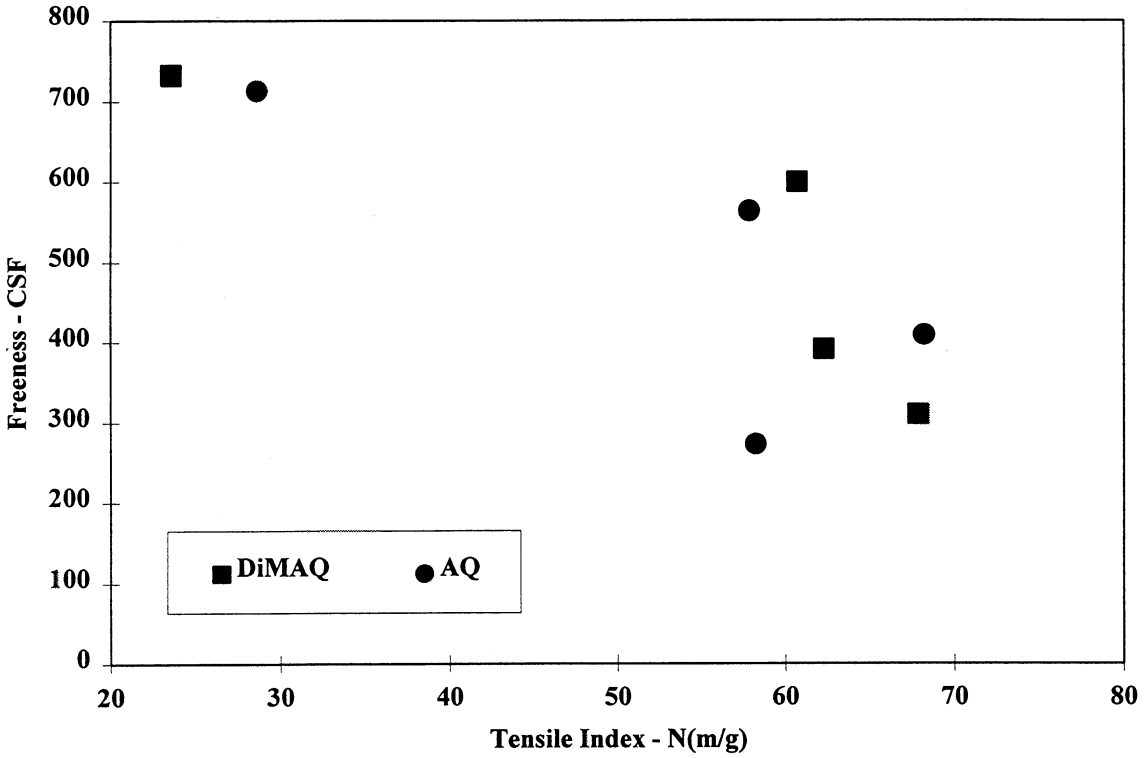

Figure 7. Comparison of Average Tensile Indexes for Two Soda-Catalyst Pulps at Four Freenesses.

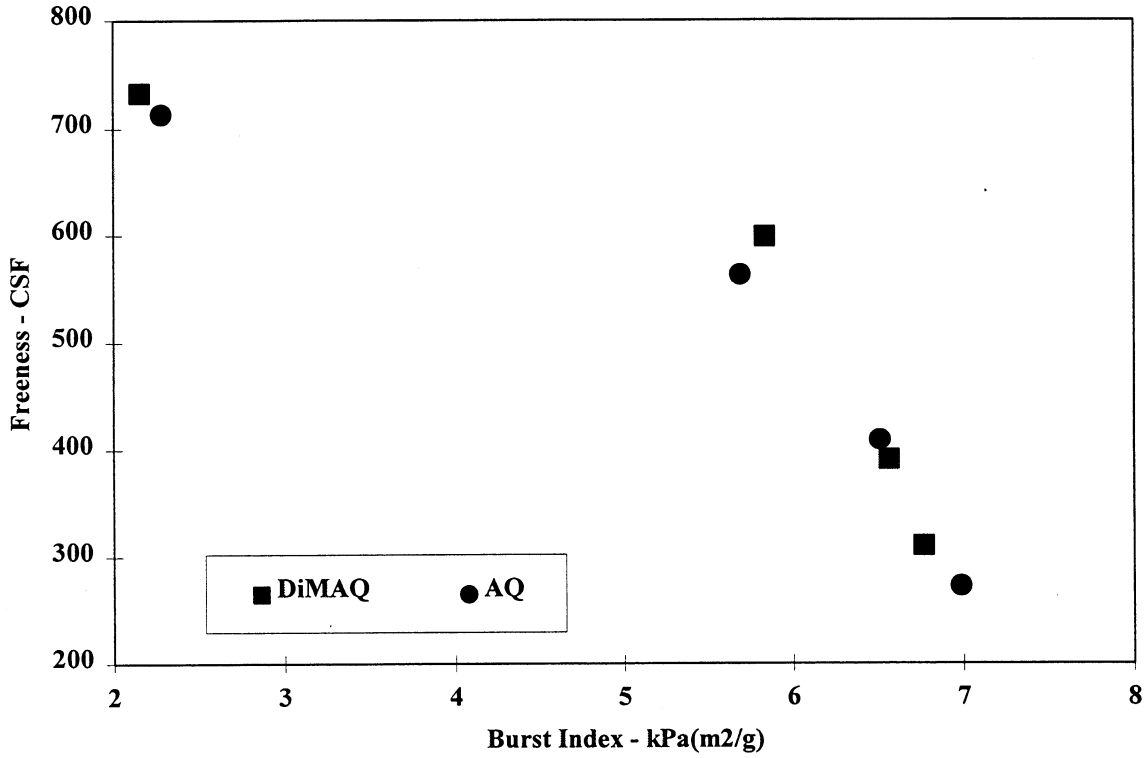

Figure 8. Comparison of Average Burst Indexes for Two Soda-Catalyst Pulps at Four Freenesses.

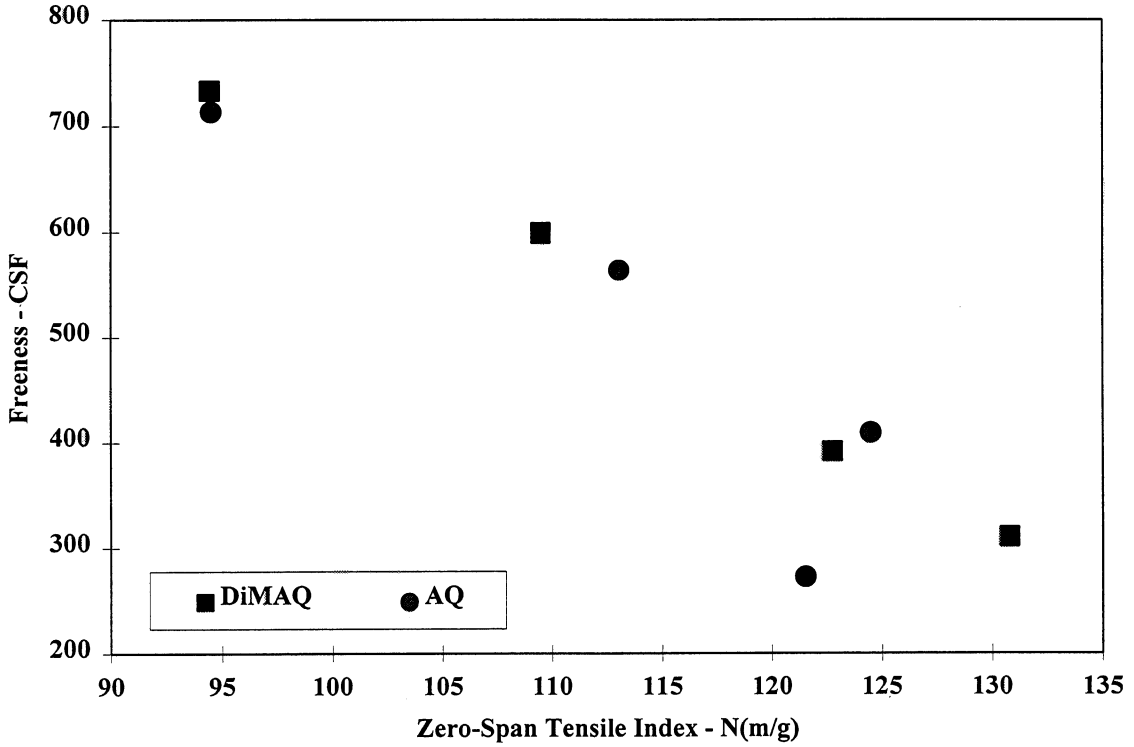

Figure 9. Comparison of Average Zero-Span Tensile Indexes for Two Soda-Catalyst Pulps at Four Freenesses.

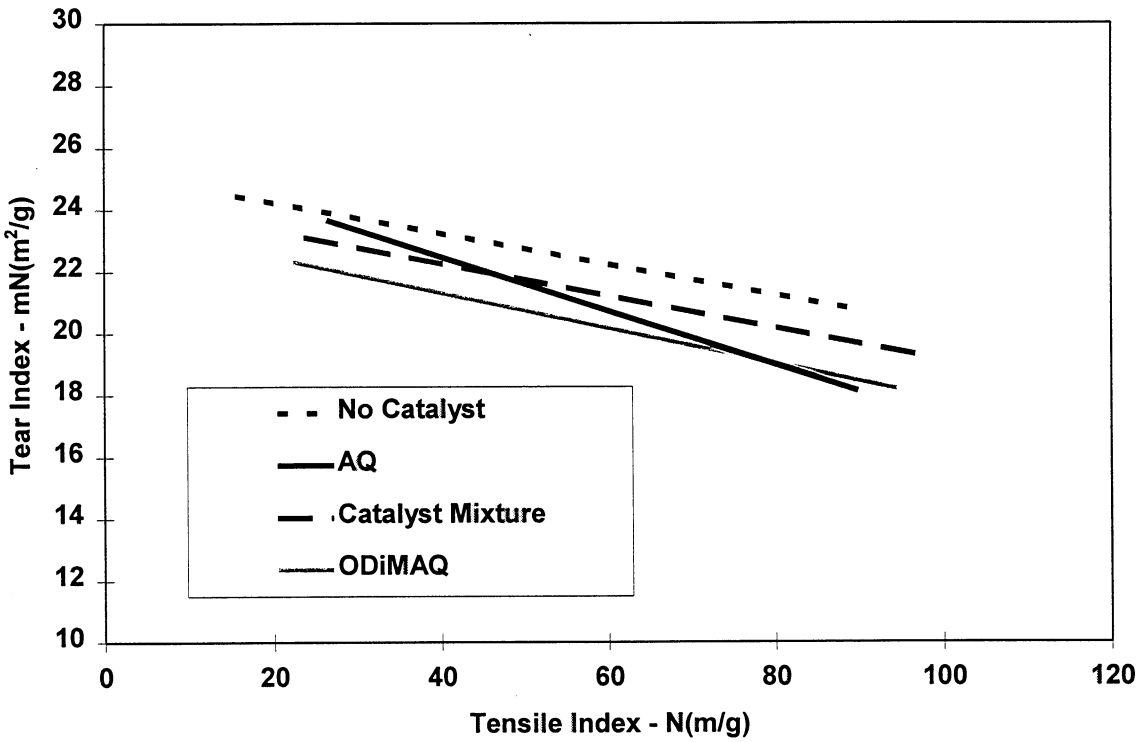

Figure 10. Comparison of Tear/Tensile Indexes Trendlines for Kraft and Kraft-Catalyst Bleached Pulps.